



PATENT SPECIFICATION

NO DRAWINGS

929,394

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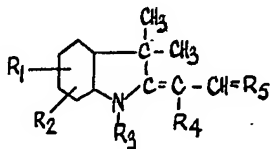
COMPLETE SPECIFICATION

Process for Dyeing or Printing Synthetic Polymers

We, FARBENFABRIKEN BAYER ARTIEN-GESELLSCHAFT, a body corporate organised under the laws of Germany, of (22 c) Leverkusen-Bayerwerk, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention is concerned with a process for dyeing and printing synthetic materials.

In accordance with the present invention, we have found that valuable dyeings and prints on materials, such as fibres, filaments and ribbons, made from synthetic polyamides and polyurethanes, as well as from aromatic polyesters, preferably polyethylene terephthalates, are obtained if there is used, as dyeing components, dyestuffs of the general formula:—



in which R₁ stands for a hydrogen, chlorine or bromine atom or a lower alkyl, lower alkoxy, β-hydroxyethoxy, amino, acetylamino, benzoylamino, nitro, cyano, sulphonamido, lower alkyl substituted sulphonamido, lower alkyl sulphonyl, lower alkyl sulphonylamino, phenyl sulphonylamino, phenyl carboxylic acid lower alkyl ester group, or —CH=CH—CH=CH— which is linked to adjacent carbon atoms of the carbocyclic nucleus of the indoline, R₂ is a hydrogen atom or a lower alkyl radical, R₃ stands for hydrogen or lower alkyl, R₄ denotes a hydrogen atom or a cyano, acyl or carboxylic acid lower alkyl ester radical, and R₅ stands for a radical of the 5-pyrazolone series linked to the 4-
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position, or a radical of cyanoacetic acid, cyanoacetic acid lower alkyl ester, cyanoacetic acid substituted lower alkyl ester such as cyanoacetic acid cyano- or hydroxy lower alkyl ester, cyanoacetic acid amide, optionally substituted in the amide group, bound in

α-position to the $\text{—}\overset{\text{O}}{\parallel}\text{C—}$ group or malonic acid dinitrile, the radical R₅ being free of sulphonic acid groups and the lower alkyl and lower alkoxy radicals containing 1 to 5 carbon atoms.

The dyestuffs are applied to the above-mentioned materials according to methods which are known as such, for instance, from an aqueous dispersion at temperatures of between about 60°C. to about 100°C. The dyestuffs can also be used for dyeing in the so-called spin dyeing process by adding them to a spinning solution before spinning. The dyeings and prints which are obtained by the improved process of the present invention are distinguished by brilliancy and excellent fastness to light.

The dyestuffs which are used in the improved dyeing and printing process according to the present invention are obtainable, for instance, by the process described and claimed in our Specification Nos. 28738/62 and 913/63 (Serial Nos. 929393 and 929395).

The following Examples are given for the purpose of illustrating the present invention, the parts by weight being grams and the parts by volume being millilitres, unless otherwise stated.

EXAMPLE 1

20 parts by weight of the dyestuff obtained from 1,3,3 - trimethyl - 5 - carbomethoxy - 2 - methylene - indoline - ω - aldehyde and cyanoacetic acid ethyl ester by the method described in Example 1 of Application No. 913/63 (Serial No. 929395) are kneaded with 80 parts by weight of a condensation product of formaldehyde and a naphthalene sulphonic

acid and with little water, until fine division is obtained. The paste is then dried at 50°C. *in vacuo*.

- 5 Yarns made from ϵ -caprolactam is introduced at 50°C. into a dye-bath which contains, per litre, 0.66 grams of the dyestuff dispersion described above, and 0.5 grams of a condensation product of formaldehyde and a naphthalene sulphonic acid. The liquor-to-goods ratio is 35 to 1. The dye-bath is heated to 100°C. within 30 minutes and dyeing continued at this temperature for one

hour. The yarn is subsequently rinsed with water and dried. A greenish-yellow shade with excellent fastness to light is obtained.

Similar shades of very good fastness properties are obtainable if the dyestuff of this Example is dyed in known manner on polyurethane fibres from hexamethylene diisocyanate and 1,4-dibutylene glycol.

Similar results are obtainable if, instead of the above-mentioned dyestuff, dyestuffs are used made from the components listed in the following Table:—

Aldehyde component	Methylene compound	m.p. of the dyestuff	Shade of the dye on polyamide fibres
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	cyanoacetic acid-ethyl ester	154—156°C.	greenish-yellow
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	cyanoacetic acid-methylamide	248—249°C.	greenish-yellow
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	cyanoacetic acid-dimethylamide	158—159°C.	greenish-yellow
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	cyanoacetic acid-butylamide	185—188°C.	greenish-yellow
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	cyanoacetic acid-piperidide	152—154°C.	greenish-yellow
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	cyanoacetic acid-benzylamide	184—187°C.	greenish-yellow
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	cyanoacetic acid-anilide	254—257°C.	greenish-yellow
1,3,3-trimethyl-5-carbomethoxy-2-methylene indoline- ω -aldehyde	cyanoacetic acid-butyl ester	165—167°C.	greenish-yellow
1,3,3-trimethyl-5-carbomethoxy-2-methylene indoline- ω -aldehyde	cyanoacetic acid- β -hydroxyethylamide	280—282°C.	greenish-yellow
1,3,3-trimethyl-5-carbomethoxy-2-methylene indoline- ω -aldehyde	cyanoacetic acid- γ -methoxy propylamide	180—182°C.	greenish-yellow
1-ethyl-3,3-dimethyl-5-carboethoxy-2-methylene-indoline- ω -aldehyde	cyanoacetic acid-ethyl ester	160—163°C.	greenish-yellow
1,3,3-trimethyl-7-phenyl-2-methylene indoline- ω -aldehyde	cyanoacetic acid-ethyl ester	197—198°C.	greenish-yellow
1,3,3-trimethyl-5-chloro-2-methylene indoline- ω -aldehyde	cyanoacetic acid-ethyl ester	208—209°C.	greenish-yellow
1,3,3-trimethyl-5-methoxy-2-methylene indoline- ω -cyan- ω -aldehyde	cyanoacetic acid-ethyl ester	191—198°C.	greenish-yellow
1,3,3-trimethyl-5-methoxy-2-methylene indoline- ω -cyan- ω -aldehyde	cyanoacetic acid-butyl ester	185°C.	greenish-yellow
1,3,3-trimethyl-5-methoxy-2-methylene indoline- ω -cyan- ω -aldehyde	cyanoacetic acid- β -cyanoethyl ester	167—174°C.	greenish-yellow

Aldehyde component	Methylene compound	m.p. of the dyestuff	Shade of the dye on polyamide fibres
1,3,3-trimethyl-2-methylene indoline- ω -aldehyde	malonic acid dinitrile	254—255°C.	greenish-yellow
1,3,3-trimethyl-2-methylene indoline- ω -aldehyde	cyanoacetic acid- β -cyano-ethyl ester	144—149°C.	greenish-yellow
1,3,3-trimethyl-5-carbomethoxy-2-methylene indoline- ω -aldehyde	malonic acid dinitrile	152—155°C.	greenish-yellow
1,3,3-trimethyl-(6,7)-benzo-2-methylene indoline- ω -aldehyde	cyanoacetic acid-ethyl ester	163—168°C.	greenish-yellow
1,3,3,5-tetramethyl-2-methylene indoline- ω -aldehyde	cyanoacetic acid-ethyl ester	170—172°C.	greenish-yellow
1,3,3-trimethyl-5-methoxy-2-methylene indoline- ω -aldehyde	cyanoacetic acid-ethyl ester	193—195°C.	greenish-yellow
1,3,3-trimethyl-2-methylene-indoline- ω -cyan- ω -aldehyde	cyanoacetic acid-ethyl ester	223—225°C.	greenish-yellow
1,3,3-trimethyl-5-carbomethoxy-2-methylene indoline- ω -aldehyde	cyanoacetic acid- β -cyano-ethyl ester	215—218°C.	greenish-yellow
1,3,3-trimethyl-5-methoxy-2-methylene indoline- ω -cyan- ω -aldehyde	malonic acid dinitrile	213—215°C.	greenish-yellow
1,3,3-trimethyl-5-methoxy-2-methylene indoline- ω -cyan- ω -aldehyde	cyanoacetic acid-anilide	225—226°C.	greenish-yellow
1,3,3-trimethyl-5-methoxy-2-methylene indoline- ω -cyan- ω -aldehyde	cyanoacetic acid-methyl ester	223—225°C.	greenish-yellow
1,3,3-trimethyl-2-methylene indoline- ω -cyan- ω -aldehyde	malonic acid dinitrile	276—277°C.	greenish-yellow
1,3,3-trimethyl-2-methylene-indoline- ω -cyan- ω -aldehyde	cyanoacetic acid-methyl ester	223°C.	greenish-yellow
1,3,3-trimethyl-5-chloro-2-methylene indoline- ω -cyan- ω -aldehyde	malonic acid dinitrile	291—292°C.	greenish-yellow
1,3,3-trimethyl-5-chloro-2-methylene indoline- ω -cyan- ω -aldehyde	cyanoacetic acid-ethyl ester	195—197°C.	greenish-yellow
1,3,3-trimethyl-5-chloro-2-methylene indoline- ω -cyan- ω -aldehyde	cyanoacetic acid-anilide	234—236°C.	greenish-yellow
1,3,3,5-tetramethyl-2-methylene indoline- ω -cyan- ω -aldehyde	malonic acid dinitrile	252—253°C.	greenish-yellow
1,3,3,5-tetramethyl-2-methylene indoline- ω -cyan- ω -aldehyde	cyanoacetic acid-ethyl ester	230—231°C.	greenish-yellow
1,3,3-trimethyl-5-carbomethoxy-2-methylene indoline- ω -cyan- ω -aldehyde	malonic acid dinitrile	227—229°C.	greenish-yellow

EXAMPLE 2

The dyestuff obtained from 1,3,3 - trimethyl - 5 - carbomethoxy - 2 - methylene indoline - ω - aldehyde and 1 - phenyl - 3 - methyl - 5 - pyrazolone by the method described in Example 2 of Specification No. 913/63 (Serial No. 929395) is applied to polyamide fibres according to the instructions given in Example 1; an exhaustive yellowish-orange dyeing of excellent fastness to light and wet processing is thus obtained. 10

Similar results are obtainable if, instead of the above dyestuff, dyestuffs are used which are produced from the starting components indicated below:— 15

Aldehyde component	Methylene compound	m.p. of the dyestuff	Shade of the dye on polyamide fibres
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	1-phenyl-3-methyl-5-pyrazolone	213—214°C.	yellowish-orange
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	3-methyl-5-pyrazolone	248—249°C.	yellowish-orange
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	1-phenyl-5-pyrazolone-3-carboxylic acid ethyl ester	217—218°C.	yellowish-orange
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	1-phenyl-5-pyrazolone-3-carboxylic acid (Na-salt)	—	yellowish-orange
1,3,3-trimethyl-5-carbomethoxy-2-methylene-indoline- ω -aldehyde	3-methyl-5-pyrazolone	266—270°C.	yellowish-orange
1,3,3-trimethyl-5-carbomethoxy-2-methylene-indoline- ω -aldehyde	1-phenyl-5-pyrazolone-3-carboxylic acid (Na-salt)	—	yellowish-orange
1,3,3-trimethyl-5-chloro-2-methylene-indoline- ω -aldehyde	1-phenyl-3-methyl-5-pyrazolone	246—249°C.	yellowish-orange
1,3,3-trimethyl-5-chloro-2-methylene-indoline- ω -aldehyde	3-methyl-5-pyrazolone	262—264°C.	yellowish-orange
1,3,3-trimethyl-7-phenyl-2-methylene-indoline- ω -aldehyde	1-phenyl-3-methyl-5-pyrazolone	218—221°C.	yellowish-orange
1,3,3-trimethyl-7-phenyl-2-methylene-indoline- ω -aldehyde	3-methyl-5-pyrazolone	232—234°C.	yellowish-orange
1,3,3-trimethyl-5-amino-2-methylene-indoline- ω -aldehyde	1-phenyl-3-methyl-5-pyrazolone	222—223°C.	reddish-orange
1-ethyl-3,3-dimethyl-5-carboethoxy-2-methylene-indoline- ω -aldehyde	1-phenyl-3-methyl-5-pyrazolone	196—198°C.	yellowish-orange
1,3,3-trimethyl-5-nitro-2-methylene-indoline- ω -aldehyde	1-phenyl-3-methyl-5-pyrazolone	above 300°C.	orange
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	1-(4 ¹ -methylphenyl)-3-methyl-5-pyrazolone	198—200°C.	yellowish-orange
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	1-(3 ¹ -chlorophenyl)-3-methyl-5-pyrazolone	223—224°C.	yellowish-orange
1,3,3-trimethyl-2-methylene-indoline- ω -aldehyde	1-(3 ¹ -nitrophenyl)-3-methyl-5-pyrazolone	294—295°C.	yellowish-orange
1,3,3,5-tetramethyl-2-methylene-indoline- ω -aldehyde	1-phenyl-3-methyl-5-pyrazolone	231—232°C.	yellowish-orange

Aldehyde component	Methylene compound	m.p. of the dyestuff	Shade of the dye on polyamide fibres
1,3,3-trimethyl-5-methoxy-2-methylene-indoline- ω -aldehyde	1-phenyl-3-methyl-5-pyrazolone	234—236 °C.	orange
1,3,3-trimethyl-5-methoxy-2-methylene-indoline- ω -aldehyde	1-(4 ¹ -nitrophenyl)-3-methyl-5-pyrazolone	268—270 °C.	orange
1,3,3-trimethyl-5-carbomethoxy-2-methylene-indoline- ω -aldehyde	1-(4 ¹ -methylphenyl)-3-methyl-5-pyrazolone	274—275 °C.	orange
1,3,3-trimethyl-5-carbomethoxy-2-methylene-indoline- ω -aldehyde	1-(2 ¹ -chlorophenyl)-3-methyl-5-pyrazolone	235—237 °C.	yellowish-orange
1,3,3-trimethyl-5-carbomethoxy-2-methylene-indoline- ω -aldehyde	1-(3 ¹ -chlorophenyl)-3-methyl-5-pyrazolone	215—217 °C.	yellowish-orange
1,3,3-trimethyl-5-carbomethoxy-2-methylene-indoline- ω -aldehyde	1-(2 ¹ ,5 ¹ -dichlorophenyl)-3-methyl-5-pyrazolone	135—138 °C.	yellowish-orange
1,3,3-trimethyl-5-carbomethoxy-2-methylene-indoline- ω -aldehyde	1-(3 ¹ -nitrophenyl)-3-methyl-5-pyrazolone	290—292 °C.	yellowish-orange
1,3,3-trimethyl-5-carbomethoxy-2-methylene-indoline- ω -aldehyde	1-(4 ¹ -aminophenyl)-3-methyl-5-pyrazolone	149—152 °C.	yellowish-orange
1,3,3-trimethyl-5-carbomethoxy-2-methylene-indoline- ω -aldehyde	1-(3 ¹ -aminophenyl)-3-methyl-5-pyrazolone	137—142 °C.	yellowish-orange
1,3,3-trimethyl-5-carbomethoxy-2-methylene-indoline- ω -aldehyde	1-(3 ¹ -acetylaminophenyl)-3-methyl-5-pyrazolone	289—290 °C.	orange
1,3,3-trimethyl-5-carbomethoxy-2-methylene-indoline- ω -aldehyde	1-(3 ¹ -carboxyphenyl)-3-methyl-5-pyrazolone	255—256 °C.	yellowish-orange
1,3,3-trimethyl-5-carbomethoxy-2-methylene-indoline- ω -aldehyde	1-phenylpyrazolone-3-carboxylic acid-amide	275—276 °C.	reddish-orange
5-nitro-1,3,3,7-tetramethyl-2-methylene-indoline- ω -aldehyde	1-phenyl-3-methyl-5-pyrazolone	257—258 °C.	orange

EXAMPLE 3

Fibres of polyethylene terephthalate are dyed in a dye-bath having a liquor-to-goods ratio of 30 to 1, and containing per litre 5 grams *o*-cresotic acid methyl ester and 1.3 grams of a dispersion of the dyestuff prepared from 1,3,3-trimethyl-5-methoxy-2-methylene-indoline - ω - cyan - ω - aldehyde and cyanoacetic acid β - cyanoethyl ester by the method described in Example 2 of Application No. 28,738/59 (Serial No. 929393) and converted into a finely divided state by the method indicated in Example 1; the pH value of the dye-bath is adjusted to 4.5 by the addition of sulphuric acid. Within 20 minutes, the initial temperature of 50°C. is increased to 100°C. and dyeing continued at this temperature for one hour. The dyed material is then rinsed with water and dried. A greenish-

yellow shade having very good fastness to light is thus obtained.

A similar result is obtained if, instead of the dyestuff indicated above, the dyestuff is used which is prepared by alkaline condensing 1,3,3 - trimethyl - 5 - methoxy - 2 - methylene - indoline - ω - cyan - ω - aldehyde and cyanoacetic acid ethyl ester.

EXAMPLE 4

A dyestuff obtained from 1 - phenyl - 3 - methyl - 5 - pyrazolone - 4 - aldehyde and 1,3,3 - trimethyl - 5 - methoxy - 2 - methylene - ω - cyanindoline by the method described in Example 3 of Application No. 28,738/59, (Serial No. 929393) when applied to polyethylene terephthalate fibres by the method described in Example 3 gives yellowish-orange shades.

Yellowish-orange shades are also produced

on polyethylene terephthalate fibres when using the dyestuff prepared from 1 - phenyl - 3 - methyl - 5 - pyrazolone - 4 - aldehyde and 1,3,3 - trimethyl - 2 - methylene - ω - cyanindoline.

Furthermore, the dyestuff obtained from 1 - phenyl - 3 - methyl - 5 - pyrazolone - 4 - aldehyde and 2,3,3 - trimethylindolenine dyes polyamide fibres in a yellowish-orange shade.

EXAMPLE 5

When using the dyestuffs obtained from the components listed in the following Table by the method described in Example 3 of Application No. 913/63 (Serial No. 929395), dyeings are obtained by the method of Example 1, on polyamide fibres in the indicated shades:—

Pyrazolone component	Indoline component	m.p. of the dyestuff	Shade of the dye on polyamide fibres
1-(<i>p</i> -tolyl)-3-methyl-5-pyrazolone-4-aldehyde	1,3,3-trimethyl-5-carbo-methoxy-2-methylene indoline	274—275°C.	orange
1-phenyl-3-carboethoxy-4-dimethyl-aminomethylene-5-pyrazolone	1,3,3-trimethyl-5-carbo-methoxy-2-methylene indoline	261—265°C.	orange
1-phenyl-3-carboethoxy-4-dimethyl-aminomethylene-5-pyrazolone	1,3,3-trimethyl-5-methoxy-2-methylene indoline	211—212°C.	orange
1-phenyl-3-(<i>p</i> -methoxyphenyl)-4-(<i>N,N</i> -dimethylaminomethylene)-5-pyrazolone	1,3,3-trimethyl-5-chloro-2-methylene indoline	249—251°C.	yellowish-orange
1-phenyl-3-methyl-4-formyl-5-pyrazolone	1,3,3,5-tetramethyl-2-cyan-methylene indoline	250—253°C.	yellowish-orange
1-phenyl-3-methyl-4-formyl-5-pyrazolone	1,3,3-trimethyl-5-methoxy-2-cyan-methylene-indoline	211—213°C.	yellowish-orange
1-phenyl-3-methyl-5-pyrazolone-4-aldehyde	1,3,3-trimethyl-5-carbo-methoxy-2-methylene indoline	231—232°C.	yellowish-orange

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EXAMPLE 6

When using the dyestuffs obtained from the components listed in the following

Table, dyeings are obtained, by the method of Example 1, on polyamide fibres in the indicated shades:—

25

Pyrazolone Component	Indoline Component	m.p. of the dyestuff	Shade of the dye on polyamide fibres
1-phenyl-3-methyl-5-pyrazolone-4-aldehyde	1,3,3-trimethyl-2-cyan-methylene-indoline	215—220°C.	yellowish-orange
1-phenyl-3-methyl-4-formyl-5-pyrazolone	1,3,3,5-tetramethyl-2-methylene-indoline- ω -carboxylic acid ethyl ester	198—200°C.	yellowish-orange
1-phenyl-3-methyl-4-formyl-5-pyrazolone	1,3,3,5-tetramethyl-2-benzoyl-methylene indoline	220—227°C.	yellowish-orange

ATTACHED
 ABSTRACT
 of GB929394

ACCESSION NUMBER: 1964:31648 CAPLUS
 DOCUMENT NUMBER: 60:31648
 ORIGINAL REFERENCE NO.: 60:5696h, 5697a
 TITLE: Dyeing or printing synthetic polymers. Farbenfabriken Bayer A.-G.
 SOURCE: 7 pp.; Division of Brit. 929,393 (CA 60, 5680d)
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 PATENT INFORMATION:

for applicant.

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 929394		19630619	GB	
DE 1172388			DE	
US 3255204		1966	US	

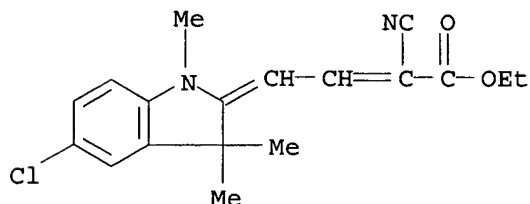
PRIORITY APPLN. INFO.: DE 19580822

AB Fibers and other materials made from synthetic polyamides, polyurethans, and aromatic polyesters can be given dyeings and prints distinguished by brilliancy and excellent fastness to light by applying the methine dyes of Brit. 929,393, Brit. 929,395 (loc. cit.). Thus, a paste was made by dispersing 20 parts of the dye prepd. from 1,3,3-trimethyl-5-carbomethoxy-2-methyleneindoline-.omega.-aldehyde and NCCH₂CO₂Et with 80 parts of a condensation product (I) of HCHO and a naphthalenesulfonic acid and with a little H₂O and drying in vacuo at 50.degree.. Yarn made from .epsilon.-caprolactam was dyed greenish yellow by introducing it at 50.degree. into a dyebath contg. 0.66 g. per l. of the dye dispersion and 0.5 g. I, with a liquor-to-goods ratio of 35: 1, heating to 100.degree. in 30 min. and continuing for 1 hr., rinsing the yarn with H₂O, and drying.

IT 95125-53-4, .DELTA.2,.gamma.-Indolinecrotonic acid, 5-chloro-.alpha.-cyano-1,3,3-trimethyl-, ethyl ester 95277-55-7, .DELTA.2,.gamma.-Indolinecrotonic acid, .alpha.-cyano-5-methoxy-1,3,3-trimethyl-, ethyl ester 95316-60-2, .DELTA.2,.gamma.-Indolinecrotonic acid, .alpha.-cyano-1,3,3,5-tetramethyl-, ethyl ester 96000-62-3, .DELTA.2,.gamma.-Indolinecrotonic acid, .alpha.-cyano-1,3,3-trimethyl-, ethyl ester (prepn. of)

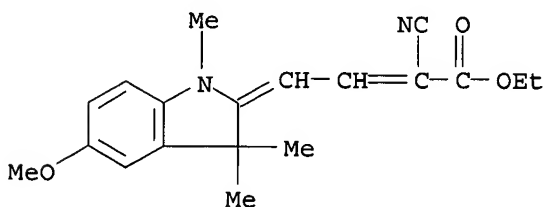
RN 95125-53-4 CAPLUS

CN .DELTA.2,.gamma.-Indolinecrotonic acid, 5-chloro-.alpha.-cyano-1,3,3-trimethyl-, ethyl ester (7CI) (CA INDEX NAME)



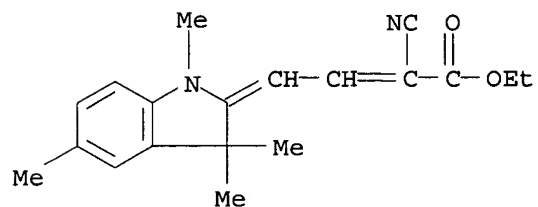
RN 95277-55-7 CAPLUS

CN .DELTA.2,.gamma.-Indolinecrotonic acid, .alpha.-cyano-5-methoxy-1,3,3-trimethyl-, ethyl ester (7CI) (CA INDEX NAME)



RN 95316-60-2 CAPLUS

CN .DELTA.2,.gamma.-Indolinecrotonic acid, .alpha.-cyano-1,3,3,5-tetramethyl-, ethyl ester (7CI) (CA INDEX NAME)



RN 96000-62-3 CAPLUS

CN 2-Butenoic acid, 2-cyano-4-(1,3-dihydro-1,3,3-trimethyl-2H-indol-2-ylidene)-, ethyl ester (9CI) (CA INDEX NAME)

